R & D STATUS REPORT

DARPA ORDER NO: 8100 **PROGRAM CODE NO:** DO-C9

CONTRACTOR: David Sarnoff Research Center

CONTRACT NO.: N00014-91-C-0216 **CONTRACT AMOUNT:** $1,585,150

EFFECTIVE DATE OF CONTRACT: 26 August 1991

EXPIRATION DATE OF CONTRACT: 25 August 1994

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SHORT TITLE OF WORK: High Performance YBCO Films

REPORTING PERIOD: 5/25/94 to 8/25/94

1 The views and conclusions contained in this document are those of the authors and should not be interpreted as necessarily representing the official policies, either expressed or implied, of the Defense Advance Research Projects Agency of the U.S. Government.

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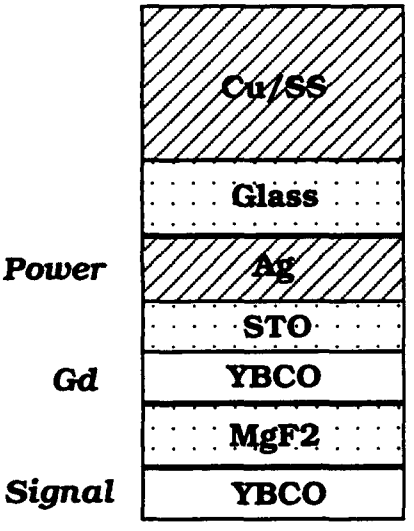
SUMMARY

- High quality YBCO films were successfully deposited on 2.5 cm by 2.5 cm magnesium fluoride substrates. Critical temperatures of 88-89K and transition widths of 0.5K were obtained.
- Surface quality of Sarnoff-polished magnesium fluoride was improved to a level that produced a χ-min of about 17% using Rutherford Backscattering.
- The maximum STO dielectric layer thickness (for capacitor between ground plane and power plane layers) was found to be 2000 angstroms or less in order to allow re-oxygenation of underlying YBCO ground plane.
- A new bismuth oxide based glass for bonding of the YBCO sample to the metal handle was identified. This glass showed significantly less interaction with silver and better stability at 750K under vacuum than the previously used PbO-based glass.
- A five micron thick silver layer for the power plane was found sufficient for preventing any reduction of its conductivity due to interaction with the bonding glass during firing.
- Patterned glass/metal handle attachment to YBCO samples showed good adhesion.

I. INTRODUCTION

An MCM technology based on a YBCO/bulk magnesium fluoride microstrip structure and attached to a glass/metal handle is being developed. Figure 1 is a cross-sectional view of the various layers in the desired MCM configuration. During this past quarter, emphasis has been placed on the following problems: (1) polishing the MgF_2 to a surface condition suitable for growing high quality YBCO films; (2) optimizing STO and silver capping layers to shield the YBCO layers from the very reactive glass/metal handle; (3) perfecting glass/metal handle attachment that has good adhesion at cryogenic temperatures and withstands the harsh environment imposed during the 750°C partial oxygen-pressured vacuum deposition (PPD). The following report shows some very promising results relating to all of these areas.



Goal structure for this proposed effort.

Figure 1. MCM/YBCO Structure Using Ag Cap Layer.

II. PROCESSING DEVELOPMENT OF MCM STRUCTURE

A. Summary of Development Plan

As shown in the last quarterly report the diagram showing the breakdown of tasks for fabricating the MCM structure is given in Figure 2 with Tasks I and II running in parallel. Detailed descriptions of the tasks are given in the Program Plan for Phase III dated November 30, 1993, that was submitted to ARPA.

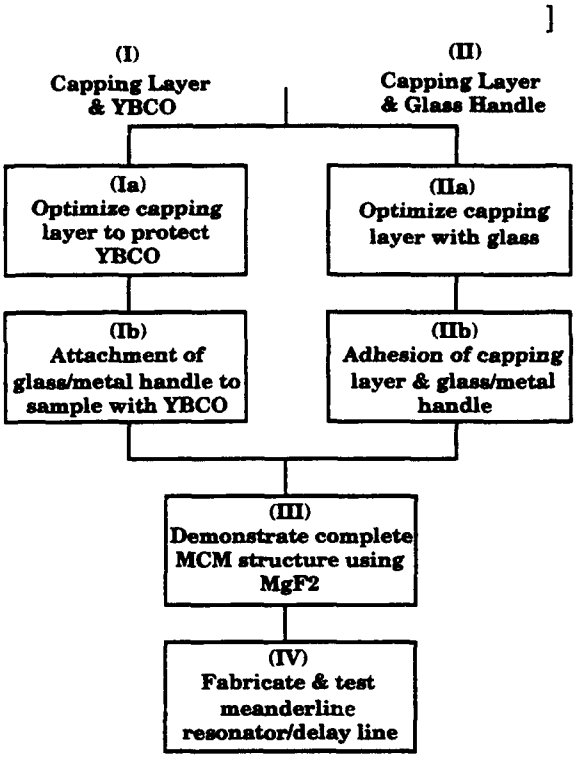


Figure 2. *Breakdown of Program Tasks.*

B. MgF_2 Substrate Processing

Work has continued in the area of polishing MgF_2 substrates. Two side polished MgF_2 substrates are needed for this program because the YBCO will be deposited on both sides of the substrate. Commercial Crystal Labs offers one side only polished samples. Samples were obtained from this vendor and the polished side marked. Sarnoff polished the other side. Average surface roughness measurements done on the samples showed the Sarnoff side to be equal to the vendor side. χ-min measurements show the Sarnoff polish has improved from the previous samples tested last quarter. The results from two of the samples are shown in Figures 3 and 4. From Figure 3, the vendor side polish produced a minimum yield of 5% while the Sarnoff polish gave 17%. From Figure 4, the vendor side is 9% and the Sarnoff side is 19%. Although the Sarnoff polish did not produce the same low ion channeling yield as the vendor polish, these results are an improvement over last quarter's reported results of 25%. Because the average surface roughness measurements indicated that the polishes were similar, we annealed the samples at 500°C in vacuum to improve the surface finish. We are awaiting χ-min test results being done at Neocera.

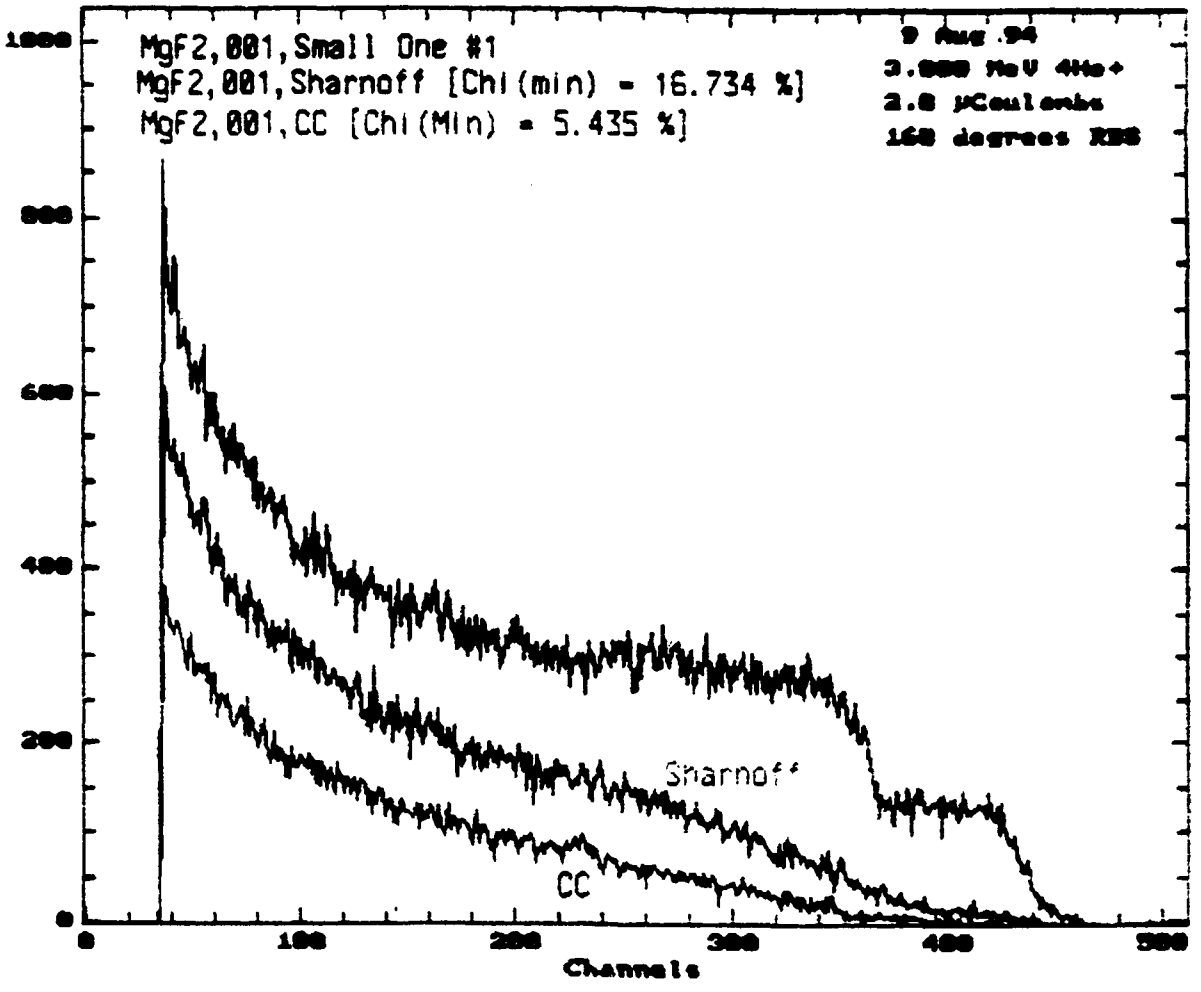


Figure 3. χ-min analysis of MgF_2 Sample.

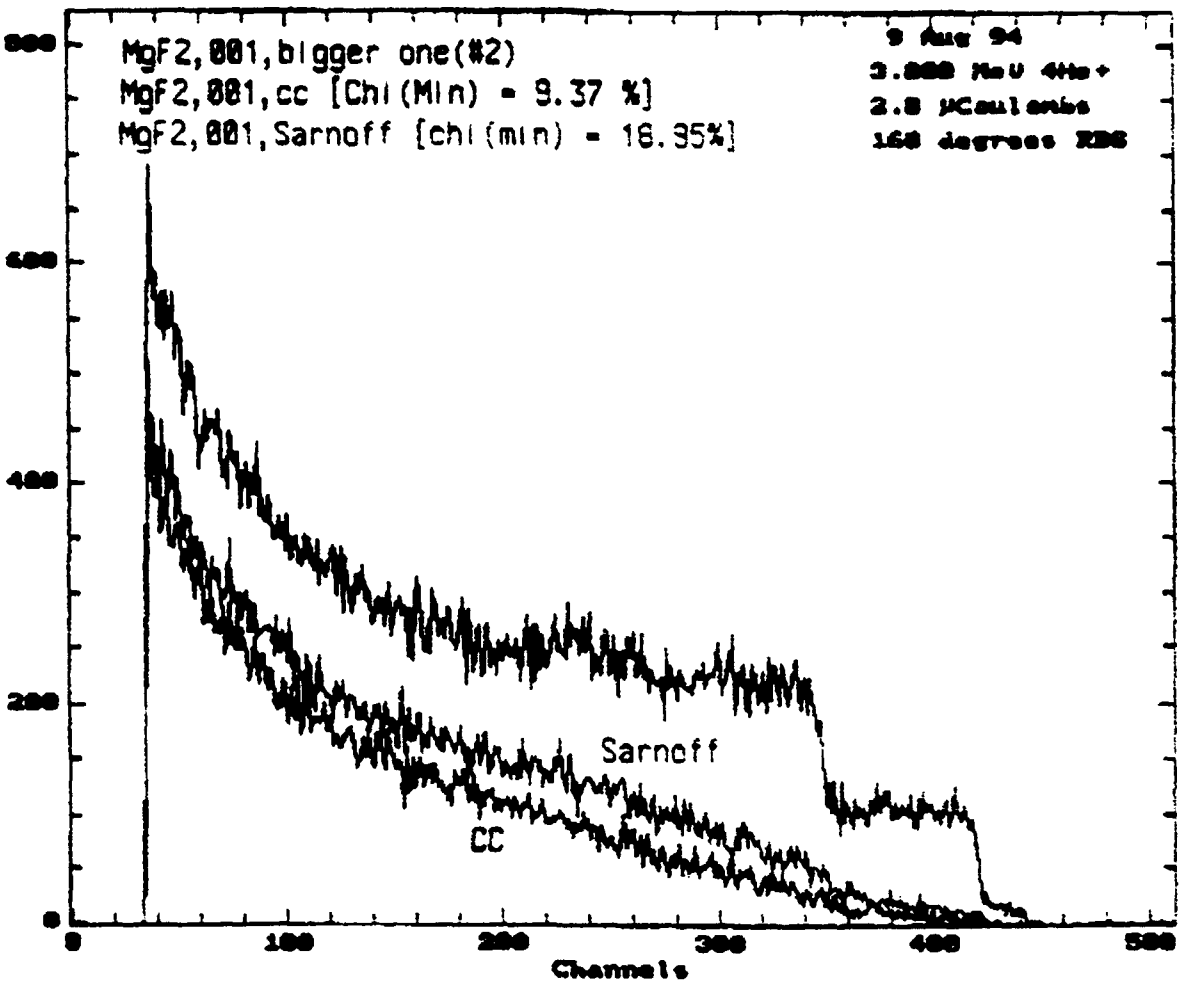


Figure 4. *χ-min analysis of MgF_2 Sample.*

C. Glass/Metal Handle Processing

1. Glass Material Considerations

Based on the results from the glass tests at 750°C in vacuum, we have chosen the Bi_2O_3-glass to use with the metal handle. Tests performed last quarter on PbO-glass and Bi_2O_3-glass in vacuum at elevated temperatures were repeated with the same results; the Bi_2O_3-glass did not blister or flow at 750°C. It was determined last quarter that 5μm of Ag was adequate to prevent any reduction of the silver's conductivity due to interaction with the bonding glass during firing.

2. Optimization of the STO Dielectric and the Ag Capping Layers

Three STO/YBCO/$LaAlO_3$ samples, NGL1, NGL2, and NGL3 received last quarter were tested during this quarter. These samples have 4000Å of STO to protect the YBCO. Annealing tests on the samples showed that oxygen annealing for any length of time, 1 hour or 18 hours, was destroying the YBCO. The dielectric resonator test set was used to measure the Q_0 value before and after annealing. Sample NGL3 had an "as received" Q_0 value of 28,323 at 77K and 24.850 GHz. After annealing for one hour at 550°C in flowing oxygen, the Q_0 dropped to 0. A further anneal for 18 hours did not revive the sample. It was felt that the STO layer was too thick and was not allowing the oxygen to penetrate to the YBCO. This would explain the other poor results obtained from sample NGL1 and NGL3. Sample NGL1 was fired in air under the glass firing conditions. The "as received" Q_0 was 30,472 at 77K and 24.876 GHz. After firing in air at 640°C, the Q_0 value dropped to 0 and could not be revived after oxygen annealing for 1 hour at 550°C. Similar problems occurred with sample NGL2. Q_0 was 28,269, measured through the back. After depositing 5μm Ag, the Q_0 dropped by 29% to 20,112. However, after annealing for 30 minutes at 550°C in oxygen, Q_0 dropped to 6007. Annealing for 5 hours and then 12 hours did not significantly improve the results. The Q_0 value of 6007 is due to the Ag. After the 30 minutes of annealing, the YBCO layer was apparently dead.

New samples NGL7, NGL9, and NGL9A were produced with a thinner STO cap layer of 2000Å to facilitate the flow of oxygen. Figure 5 illustrates typical layer thicknesses. Sample NGL7 had an as received Q_0 value of 24,982. After annealing for 12 hours, Q_0 dropped by 5% to 23,850. A further anneal for 18 hours did not change Q_0 by more than 1%. We feel that this is within the accuracy of the measurement system. This is very encouraging considering that sample NGL3 with a 4000Å STO cap layer was annealed and the sample died. Sample NGL9

had an as received Q_0 value of 22,481. This sample was fired in air using the glass firing profile causing Q_0 to drop by 33% to 14,968. The sample was annealed in flowing oxygen for 18 hours. This annealing returned the Q_0 to its' original value. This result is very significant. It shows that a sample which has been heated in air to over 550°C can be revived by annealing in oxygen.

Five microns of Ag was deposited on sample NGL9A. Q_0 before the deposition was 30,095, which was measured through the back of the sample with the dielectric resonator test set. After the Ag deposition, Q_0 was measured at 16,980, a drop of 44%. This is slightly worse than the sample with the thicker buffer layer where Q_0 dropped 29%. Annealing sample NGL9A for 18 hours did not improve Q_0; it dropped to 50% of the original value. It is most likely that a much longer annealing time is needed for the oxygen to get through the Ag layer.

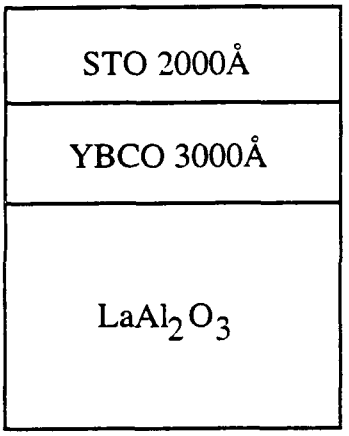


Figure 5. *YBCO samples with STO Cap Layer*

In an effort to eliminate post annealing after the Ag deposition, we changed from sputter depositing the Ag to thermal evaporation. Thermal evaporation is a far less energetic method of deposition than sputtering and would therefore minimize the damage to the YBCO. Five microns of Ag were deposited on another STO/YBCO/$LaAl_2O_3$ sample NGL10 using a thermal evaporator. Q_0 dropped by only 20%. A thermocouple has been placed in the evaporator to monitor the temperature during the Ag deposition. For a 1μm test run the maximum temperature was 35°C. We will do another run at 5μm and monitor the temperature. The results from all of the $LaAl_2O_3$ samples are summarized in Table 1.

Two STO/YBCO/MgF_2 samples, NCF54 and NCF55, were provided by Neocera and tested in the dielectric resonator test set. The Q_0 values were only 16,204 and 17,700, respectively. The Tc values for both samples were 88K and ΔTc =

Table 1
STO/YBCO/LaAlO3 Samples

Sample	Sub	STO (Å)	Ag (um)	Qo (front)	f (GHz) (front)	Qo (back)	f (GHz) (back)	Status	Qo Δ from orginal
NGL1	LaAlO3	4000	-	30472	24.876	33546	22.651	As Recd.	-
NGL1	LaAlO3	4000	-	0		-	-	Air firing, 640°C	-100%
NGL1	LaAlO3	4000	-	0		-	-	O2 anneal 1 hr.	
NGL1	LaAlO3	4000	-			-	-	O2 anneal 18 hr.	
NGL2	LaAlO3	4000	-	24422	24.875	28269	22.646	As Recd.	-
NGL2	LaAlO3	4000	5/S	-	-	20112	22.633	after sputtering Ag	-29%
NGL2	LaAlO3	4000	5/S	-	-	6007	22.633	O2 anneal 0.5 hr.	
NGL2	LaAlO3	4000	5/S	-	-	8264	22.635	O2 anneal 5 hr.	
NGL2	LaAlO3	4000	5/S	-	-	7300	22.633	O2 anneal 12 hr.	
NGL2	LaAlO3	4000	5/S	-	-			O2 anneal 18 hr.	
NGL2	LaAlO3	4000	5/S	-	-			GLASS/HANDLE	
NGL3	LaAlO3	4000	-	28323	24.850	33349	22.645	As Recd.	
NGL3	LaAlO3	4000	-	0		-	-	O2 anneal, 1 hr	
NGL3	LaAlO3	4000	-	0		-	-	O2 anneal 18 hr	
NGL7	LaAlO3	2000	-	24982	24.982	-	-	As Recd.	-
NGL7	LaAlO3	2000	-	23850	24.837	-	-	O2 anneal 12 hr	-5%
NGL7	LaAlO3	2000	-	23422	24.839	-	-	O2 anneal 18 hr	-6%
NGL9	LaAlO3	2000	-	22481	24.871	-	-	As Recd.	-
NGL9	LaAlO3	2000	-	14968	24.839	-	-	Air firing, 640°C	-33%
NGL9	LaAlO3	2000	-	22384	24.839	-	-	O2 anneal 18 hr	
NGL9A	LaAlO3	2000	-	23800	24.864	30095	21.698	As Recd. (t=0.020")	-
NGL9A	LaAlO3	2000	5/S	-	-	16980	21.699	after sputtering Ag	-44%
NGL9A	LaAlO3	2000	5/S	-	-	15046	21.690	O2 anneal 18 hr	-50%
NGL9A	LaAlO3	2000	5/S	-	-	8300	21.693	GLASS/HANDLE	
NGL9A	LaAlO3	2000	5/S	-	-	5300	21.695	GLASS/HANDLE @ 166K	
NGL10	LaAlO3	2000	-	24216	24.850	31224	21.678	As Recd.(back 8/12/94)	-
NGL10	LaAlO3	2000	5/E	-	-	23700	21.688	after Ag evaporation	-20%
NGL11	LaAlO3	2000	-	23774	24.849	-	-	As Recd.	-
NGL13	LaAlO3	2000	-	25900	24.785	-	-	As Recd.	-

Table 2
STO/YBCO/MgF2 Samples

Sample	Sub	STO (Å)	Ag (um)	Qo (front)	f (GHz) (front)	Qo (back)	f (GHz) (back)	Status	Qo Δ from orginal
NCF54	MgF2	2000	-	16204	24.849	-	-	As Recd.	-
NCF55	MgF2	2000	-	17700	24.846	-	-	As Recd.	-

0.5K. More samples with a higher Q value will be obtained from Neocera during the next quarter followed by Ag and glass handle processing by Sarnoff. These results are summarized in Table 2.

3. Glass/Metal Handle Attachment

Sample NGL2 with Ag/STO/YBCO/$LaAl_2O_3$ was used as a mechanical test sample for attachment of a glass/metal handle. Although the YBCO had totally degraded as a result of earlier tests, the sample was still usable to check the adhesion of the glass/metal handle to the Ag layer. The glass was screened on the Cu/SS/Cu handle in a checkerboard pattern to allow a path for any organics in the glass to escape during firing. The pattern is shown in Figure 6. The sample was fired in a new annealing furnace in flowing oxygen using the ramp-up profile for the glass but cooling at the YBCO annealing profile. The glass/metal handle adherence to the Ag/YBCO/$LaAlO_3$ sample was excellent. It appears that the YBCO under the glass pattern was intact and not attacked by the glass. However, YBCO not under the glass was degrading visibly. It was discovered later that the new furnace was contaminated and was destroying YBCO/$LaAlO_3$ test samples. Therefore, the degradation of the YBCO not under the glass was most likely caused by this problem.

Sample NGL9A was used for the second test. Five microns of Ag had been sputter deposited as previously mentioned. Conditions were identical to those used for the first test except the regular annealing furnace was used. The adhesion was again excellent. The YBCO did not appear degraded in any way. However, the sample's Q_0 was degraded to about 8000 when checked with a dielectric resonator test set. The glass/metal handle sample was cryogenically cooled to 77°K and mechanically withstood temperature cycling. Recovering the YBCO through the thick Ag and glass/metal handle with O_2 annealing is the next obstacle to overcome.

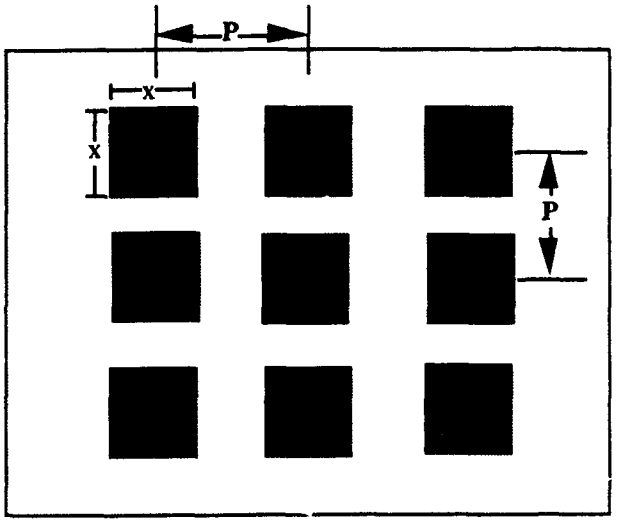


Figure 6. *Glass Pattern on Metal Handle (X=0.0508 cm, p=0.1016 cm).*

III. REFERENCES: None

IV. CHANGE IN KEY PERSONNEL: None

V. SUMMARY OF SUBSTANTIVE INFORMATION DERIVED FROM SPECIAL EVENTS: None

VI. ACTION REQUIRED BY THE GOVERNMENT: None

VII. FISCAL STATUS:

1. Amount currently provided on contract: $1275K
2. Expenditures and commitments to date: $1074K
3. Funds required to complete work: $1585K